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(54) [Title of the Invention] Solid-Liquid Separation Method

(57) [Summary]

[Object] To provide a solid-liquid separation method that has been improved so that clogging is prevented in a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation can be performed efficiently.

[Means of Achievement] A slurry containing particles of indeterminate form as solids is continuously supplied to a screen-type centrifugal separator in which a screw conveyor is disposed, and solid and liquid fractions are separated, wherein the residence time of the solid-liquid separation on the screen is set to 2 to 20 seconds, the residence time is assumed to start after the cleaning fluid passes through a supply area when a cleaning fluid supply hole is provided, and a centrifugal force of 300 to 5,000 G is applied to the screen.

[Claims]

[Claim 1] A solid-liquid separation method wherein a slurry containing particles of indeterminate form as solids is continuously supplied to a screen-type centrifugal separator in which a screw conveyor is disposed, said method characterized in that the residence time of the solid-liquid separation on the screen is set to 2 to 20 seconds, the residence time is assumed to start after a cleaning fluid passes through a supply area when a cleaning fluid supply hole is provided, and a centrifugal force of 300 to 5,000 G is applied to the screen.

[Claim 2] The solid-liquid separation method according to claim 1, wherein the screen-type centrifugal separator has an outer rotating cylinder (1), a screw conveyer (2) that is mounted inside the outer rotating cylinder while allowed to rotate in relative fashion and that comprises a cylindrical rotating shaft (21) and a screw (22), and a slurry supply pipe (3) that is disposed within the rotating shaft of the screw conveyor and that supplies a slurry to the interior of the shaft; wherein a slurry supply port (23) for supplying a slurry to the outer rotating cylinder (1) is provided on the proximal end side of the screw conveyer (2), the outer rotating cylinder (1) is sequentially constituted from a large-diameter region (11) on the proximal end side, a slanted region (12) of gradually decreasing diameter, and a small-diameter region (13) on which a screen (13a) is provided; an overflow port (4) is provided to the proximal end of the large-diameter region (11), and a solids discharge port (5) is provided at the distal end of the small-diameter region (13).

[Claim 3] The solid-liquid separation method according to claim 2, wherein the interior of the rotating shaft (21) of the screw conveyer is partitioned into a slurry supply region (21a) on the proximal end side and a cleaning fluid supply region (21b) on the distal end side; the slurry supply region (21a) is designed to be capable of supplying the slurry via the slurry supply pipe (3), and the cleaning fluid supply region (21b) is designed to be capable of supplying the cleaning fluid via a cleaning fluid supply pipe (6) inserted into the interior of the slurry supply region (3), and a cleaning fluid supply port (24) through which cleaning fluid is supplied to the small diameter region (13) of the outer rotating cylinder (1) is provided on the distal end side of the screw conveyer (2).

[Claim 4] The solid-liquid separation method according to any of claims 1 through 3, wherein a screen with an opening size that allows the passage of an amount of solids equivalent to 1 to 10 wt% in the supplied slurry is used as the screen of the screen-type centrifugal separator.

[Detailed Description of the Invention]

[0001]

[Technological Field of the Invention] The present invention relates to a solid-liquid separation method, and specifically relates to a solid-liquid separation method that uses a screen-type centrifugal separator in which a screw conveyer is disposed.

[0002]

[Prior Art] In a process for manufacturing terephthalic acid using liquid-phase oxidation of p-xylene, for example, terephthalic acid precipitates as crystals and a slurry is formed. The solids (terephthalic acid crystals) obtained as a result of such crystallization are particles of indeterminate form with a grain size distribution.

[0003] One method of solid-liquid separation for a slurry containing such particles of indeterminate form as described above is a method wherein a slurry is continuously supplied to a screen-type centrifugal separator in which a screw conveyer is disposed, and solid-liquid separation takes place on the screen due to the application of centrifugal force.

[0004] To allow the centrifugal separator to efficiently operate in such a continuous solid-liquid separation method, it is important to determine the level to which the residence time of solid-liquid separation on the screen should be set, but a sufficient proposal has not yet been made concerning these matters.

[0005]

[Problems to Be Solved by the Invention] With the foregoing aspects in view, it is an object of the present invention to provide a solid-liquid separation method that has been improved so that clogging is prevented in a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation can be performed efficiently.

[0006]

[Means Used to Solve the Above-Mentioned Problems] Specifically, the purpose of the present invention lies in a solid-liquid separation method in which a slurry containing particles of indeterminate form as solids is continuously supplied to a screen-type centrifugal separator in which a screw conveyor is disposed, and solid and liquid fractions are separated, wherein this method is characterized in that the residence time of the solid-liquid separation on the screen is

set to 2 to 20 seconds, the residence time is assumed to start after a cleaning fluid passes through a supply area when a cleaning fluid supply hole is provided, and a centrifugal force of 300 to 5,000 G is applied to the screen.

[0007]

[Embodiments of the Invention] The present invention will now be described in detail.

[0008] First, the slurry will be described. The slurry used in the present invention contains particles of indeterminate form as solids. One specific example of this slurry is one containing crude terephthalic acid crystals recovered from the step of manufacturing terephthalic acid using liquid-phase oxidation of p-xylene as previously described. This slurry is usually composed of acetic acid as a reaction solvent, terephthalic acid crystals, a catalyst dissolved in the reaction solvent, unreacted starting materials, by-products, unprecipitated terephthalic acid, and the like. Also, the average particle size of the terephthalic acid crystals is usually 120 μm $\pm$ 40 μm.

[0009] Next, a screen-type centrifugal separator in which a screw conveyer is disposed will be described. In the present invention, it is possible to use screen-type centrifugal separators having a variety of structures, regardless of their descriptive name, as long as solid-liquid separation can be performed using a screen via the action of a centrifugal force while the material to be treated is transported with a screw conveyor. Examples known in the art include a "decanter-type centrifugal separator" (JP (Kokai) 7-155643, et al.) and a "screen-bowl decanter centrifugal separator" (JP (Kokai) 2000-350946, WO 98/18750, et al.).

[0010] Fig. 1 is a cross-sectional explanatory diagram of a suitable screen-type centrifugal separator that can be used in the present invention. The screen-type centrifugal separator shown in this diagram is structured having an outer rotating cylinder (1), a screw conveyer (2) that is mounted inside the outer rotating cylinder while allowed to rotate in relative fashion and that comprises a cylindrical rotating shaft (21) and a screw (22), and a slurry supply pipe (3) that is disposed within the rotating shaft of the screw conveyor and that supplies a slurry to the interior of the shaft. The proximal end side of the screw conveyor (2) is provided with a slurry supply port (23) for supplying the slurry to the outer rotating cylinder (1). The outer rotating cylinder (1) is sequentially constituted from a large-diameter region (11) on the proximal end side, a slanted region (12) of a gradually decreasing diameter, and a small-diameter region (13) on which a screen (13a) is provided. An overflow port (4) is provided to the proximal end of the

large-diameter region (11), and a solids discharge port (5) is provided at the distal end of the small-diameter region (13).

[0011] In a preferred embodiment, the interior of the rotating shaft (21) of the screw conveyor is partitioned into a slurry supply region (21a) on the proximal end side and a cleaning fluid supply region (21b) on the distal end side. The slurry supply region (21a) is designed to be capable of supplying the slurry via the slurry supply pipe (3), and the cleaning fluid supply region (21b) is designed to be capable of supplying the cleaning fluid via a cleaning fluid supply pipe (6) inserted into the interior of the slurry supply region (3). A cleaning fluid supply port (24) through which cleaning fluid is supplied to the small-diameter region (13) of the outer rotating cylinder (1) is provided on the distal end side of the screw conveyor (2).

[0012] A plurality of openings (71c) are provided to the outer peripheral region of a flange (71) of a drive shaft (7) for the outer rotating cylinder, and these openings constitute the solids discharge port (5). Openings (81c) are provided to the outer peripheral region of a flange (81) of a drive shaft (8) for the screw conveyor to constitute the overflow port (4).

[0013] All of these elements are housed in a casing (9). The inside of the casing (9) is divided by partitioning walls into an overflow liquid reservoir (91), a filtrate reservoir (92), and a solids reservoir (93); and the filtrate reservoir (92) is further divided by partitioning walls into three compartments (92a), (92b), and (92c) along the longitudinal direction of the small-diameter region (13) for the convenience of filtrate sampling. Discharge piping is provided respectively for the overflow liquid reservoir (91); the compartments (92a), (92b), and (92c) of the filtrate reservoir (92); and the solids reservoir (93).

[0014] In the case of a screen-type centrifugal separator in which a screw conveyer is disposed, clogging is promoted by a solidified layer (cake layer) formed in a compacted state by the screw on the inside of the screen. In view of this, in the present invention, the use of a screen with openings that allow 1 to 10 wt% of the solids to pass in the slurry supplied is recommended in order to allow clogging in the screen-type centrifugal separator to be prevented and efficient solid-liquid separation to be performed.

[0015] The openings in the screen used in the present invention must be of a size capable of allowing continuous passage of, e.g., 1 to 10 wt% (preferably 1.5 to 8 wt%) of the solids (indicated as a (kg/hr)) in the slurry continuously supplied at a rate of A (kg/hr). The passage of particles through the screen openings is believed to occur preferentially with those of

indeterminate form and small diameters; in the present invention, therefore, particles of indeterminate form and relatively small diameters in the slurry are allowed to pass through the screen openings.

[0016] Consequently, the solidified layers (cake layers) formed in a compacted state in the inside of the screen, and specifically in the spaces between the outer rotating cylinder (1) and the screw (22), are constituted from particles of indeterminate form and relatively large size. The reason that excessive clogging does not occur with such solidified (cake) layers has yet to be definitively clarified, but the explanation is presumed to be as provided hereunder.

[0017] In other words, when particles of indeterminate form and relatively small size are contained in the solidified crystal (cake) layers, such cakes typically become firm and solid due to a strong bridging action caused as a result. On the other hand, solidified crystal (cake) layers composed of particles of indeterminate form and of relatively large size tend to collapse readily due to the absence of a strong bridging action. Consequently, the solidified crystal (cake) layers in the present invention are presumed to continuously collapse and rearrange, and are accordingly not believed to cause excessive screen clogging.

[0018] The clogging preventive effect cannot be achieved with screens having an opening size that causes the pass rate to be less than 1 wt%. On the other hand, with screens whose opening size allows more than 10 wt% of the particles to pass, the anticlogging effect may reach saturation while the pass rate becomes too high, thereby resulting in an inefficient operation.

[0019] The most remarkable feature of the present invention is that the residence time of solid-liquid separation on the screen is set to 2 to 20 seconds. The residence time is assumed to start after the cleaning fluid passes through the supply area when a cleaning fluid supply port is included, and a centrifugal force of 300 to 5000 G is applied to the screen.

[0020] If the centrifugal force is less than 300 G, the residence time for solid-liquid separation on the screen will be too high, and when the centrifugal force exceeds 5000 G, the centrifugal separator will become difficult to operate in a stable manner. The preferred range of centrifugal force is 500 to 3000 G. The centrifugal force is adjusted as a result of controlling the number of rotations of the motor that drives the outer rotating cylinder (1).

[0021] In the present invention, it is vital that the residence time of the solid-liquid separation (deliquoring) on the screen be set to 2 to 20 seconds at the centrifugal force described above. If the residence time is less than two seconds, solid-liquid separation will be impossible to perform

to a sufficient degree, while if the residence time exceeds 20 seconds, the solid-liquid separating effect will reach saturation, resulting in the centrifugal separator operating at reduced efficiency. The preferred residence time is three to five seconds. The residence time can be adjusted either by means of a method whereby an appropriate design is selected for the size (length) of the screen, or as a result of controlling the operating conditions whereby an appropriate difference in rotational speed is selected for the outer rotating cylinder (1) and the screw conveyor (2) (i.e., the transfer speed of the treated material across the screen).

[0022] In the present invention, the above-mentioned residence time is assumed to start after the cleaning fluid sprayed from the cleaning fluid supply port (24) passes through the supply area, when a cleaning fluid supply pipe (6)[1] is provided, as with the centrifugal separator shown in Fig. 1. Specifically, as used in the present invention, the term "residence time" refers to the time on the screen starting when only deliquoring occurs without an increase in the liquid content due to the effects of the cleaning fluid.

[0023] In the present invention, solid-liquid separation is performed as follows. The slurry is supplied from the slurry supply pipe (3) to the large-diameter region (11) of the outer rotating cylinder (1) via the slurry supply region (21a). The solids and liquids herein are then separated as a result of the centrifugal action of the outer rotating cylinder (1) rotating at a high speed.

[0024] The separated liquid is discharged from the overflow port (4) via the overflow liquid reservoir (91). The solids are moved from the slanted region (12) to the small-diameter region (13) by the screw (22). At this point, the mother liquor in the solids is separated as a result of the centrifugal force in the screen (13a). At the same time, the cleaning fluid is supplied from the cleaning fluid supply pipe (6). The cleaning fluid is sprayed from the cleaning fluid supply port (24) onto the moving solids via the cleaning fluid supply region (21b). The washed and dehydrated solids are discharged from the solids discharge port (5) via the solids reservoir (93).

[0025]

[Working Examples] The present invention is described in further detail below with the help of working examples, but the present invention is not limited to the following working examples as long as they do not deviate from the scope thereof.

[1] Translator's note: The original reads "cleaning fluid supply pipe (5)," which is probably a typographical error.

[0026] Working Examples 1-4 and Comparative Examples 1-2

A slurry containing terephthalic acid crystals (average particle size: 100 μm) obtained in the terephthalic acid manufacturing step was continuously supplied to the screen-type centrifugal separator (screen openings: 100 μm) shown in Fig. 1, and the solids and liquids were separated under the conditions shown in Table 1 (the centrifugal force on the screen was 750 G). The liquid content of the recovered cake is shown in Table 1.

[0027]

[Table 1]

	Residence time (seconds)	Slurry Supply Rate (kg/hr)	Liquid Content of Recovered Cake (wt%)
Working Example 1	3.2	1110	13.0
Working Example 2	3.2	1480	12.7
Working Example 3	5.6	1110	11.6
Working Example 4	5.6	1480	11.1
Comparative Example 1	0.8	1110	19.4
Comparative Example 2	0.8	1480	21.4

[0028] The residence time shown in Table 1 refers to the time after the slurry has passed through the cleaning fluid supply zone where the cleaning fluid is sprayed from the supply port (24) (and more specifically refers to the time after the point at which two seconds has elapsed after the slurry has passed directly under the cleaning fluid supply port (24)). The residence times and the liquid content of the recovered cakes were determined in the following manner for the sake of convenience. In other words, the filtrate specimens were recovered from the positions corresponding to the particular residence times shown above, measurements were made of the concentration of solids in the filtrate specimens for each residence time, and the liquid content of the recovered cakes was determined wit the help of material balance calculations.

[0029] As is clear from the results in Table 1, the residence times in the working examples are appropriate, so the centrifugal separator operates efficiently and a sufficiently dehydrated cake is recovered. In the comparative examples, on the other hand, the recovered cake has a high liquid content because the residence time is short.

[0030] Reference Examples

A slurry containing terephthalic acid crystals (average particle size: 100 μm) obtained in the terephthalic acid manufacturing step was continuously supplied to the screen-type centrifugal

separator shown in Fig. 1 that had a screen with the openings shown in Table 2, and the solids and liquids were separated. The results are shown in Table 2.

[0031]

[Table 2]

	Screen openings (μm)	Ratio of solids passing through screen openings to solids in supplied slurry (wt%)	Mean particle size of solids passing through screen openings (μm)	Liquid content of recovered solids (wt%)
Reference Example 1	100	3	60	11
Reference Example 2	70	0.5	15	20

[0032] As is clear from the results shown in Table 2, the solids recovered in Reference Example 1 have a low liquid content compared with Reference Example 2. This is attributable to the fact that clogging was prevented, which allowed the liquid to be satisfactory removed.

[0033]

[Effect of the Invention] As has been described in the foregoing, the present invention provides a solid-liquid separation method that has been improved so that clogging is prevented in a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation can be performed efficiently. Therefore, the present invention is of high commercial value.

[Brief Description of the Drawings]

[Figure 1] A cross-sectional illustrative diagram of a screen-type centrifugal separator that is suited for use in the present invention.

[Key]

1: outer rotating cylinder

11: large-diameter region

12: slanted region

13: small-diameter region

13a: screen

2: screw conveyer

21: rotating shaft

21a: slurry supply region

21b: cleaning fluid supply region

22: screw

23: slurry supply port

24: cleaning fluid supply port

3: slurry supply pipe

4: overflow port

5: solids discharge port

6: cleaning fluid supply pipe

7: outer rotating cylinder drive shaft

71: flange

71c: opening

8: screw conveyer drive shaft

81: flange

81c: opening

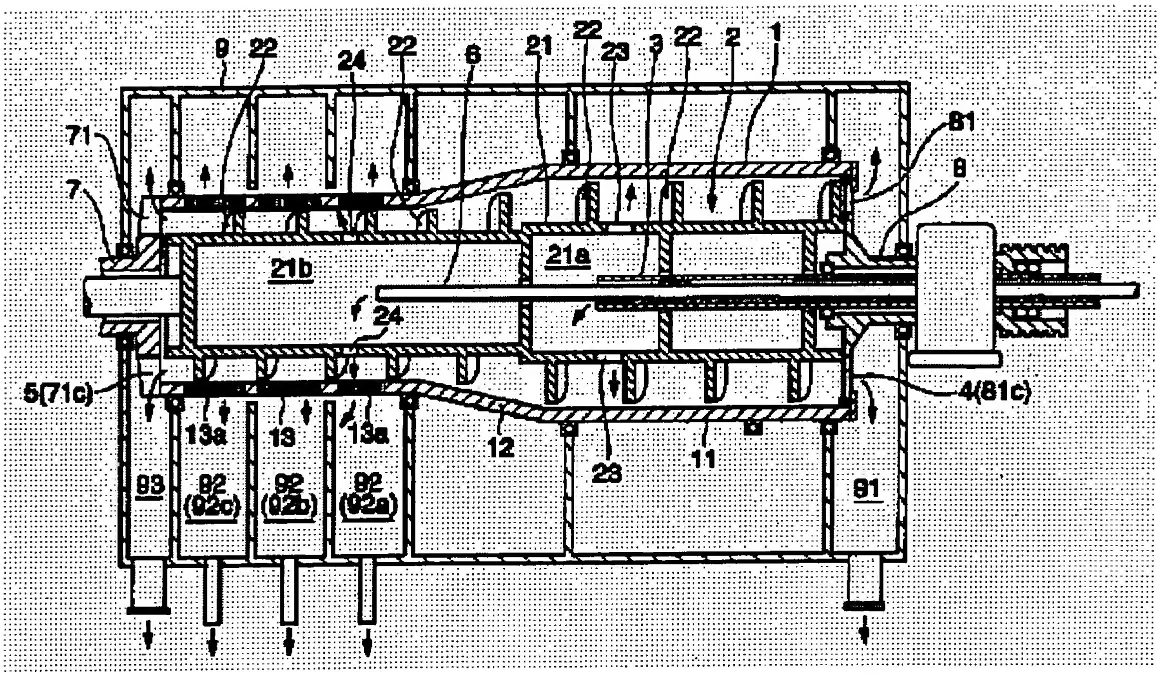
9: casing

91: overflow liquid reservoir

92: filtrate accommodating chamber

93: solids reservoir

[Fig. 1]



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